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To cite this article: Trevor P. Almeida *et al* 2017 *J. Phys.: Conf. Ser.* **903** 012022

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Preparation of high-quality planar FeRh thin films for *in situ* TEM investigations

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Abstract. The preparation of a planar FeRh thin film using a focused ion beam (FIB) secondary electron microscope (SEM) for the purpose of *in situ* transmission electron microscopy (TEM) is presented. A custom SEM stub with 45° faces allows for the transfer and milling of the sample on a TEM heating chip, whilst Fresnel imaging within the TEM revealed the presence of the magnetic domain walls, confirming the quality of the FIB-prepared sample.

1. Introduction

Equiatomic iron-rhodium (Fe₄₈Rh₅₂ to Fe₅₆Rh₄₄) has attracted much interest due to its magnetostructural transition from its antiferromagnetic (AF) to ferromagnetic (FM) phase. This ordered α' alloy adopts a CsCl structure and is AF at room temperature, but makes a first-order phase transition to a FM state at ~ 350 to 380 K, and hence can present phase co-existence and hysteresis [1]. The co-existing phases are separated by a phase-boundary domain wall (DW) and effective control over the creation and motion of these phase boundary DWs are considered desirable for potential application in a new generation of novel nanomagnetic or spintronic devices [2]. Previous studies have shown that the DWs can be nucleated in FeRh films and driven through combining heating with differential gradients of chemical doping [3]. Current understanding of the behaviour of DWs in FeRh is based upon bulk magnetic measurements and imaging, *e.g.* Kerr imaging or X-ray circular dichroism. Significant advances in knowledge are potentially enabled using the transmission electron microscopy (TEM) techniques of Fresnel imaging, off-axis electron holography and differential phase contrast imaging, which allow for the visualising of magnetic induction at nanometre spatial resolution. Further, the development of TEM holders which include micro-electro-mechanical systems (MEMS) has led to the ability to apply a range of external stimuli to samples *in situ* within the TEM, *i.e.* temperature, electrical bias, etc., whilst maintaining stability and minimising drift. TEM lamellae of bulk samples have previously been prepared on these MEMS-based chips [4] and planar magnetic thin films have been made for magnetic imaging within the TEM [5]. However, limited work has been reported on the preparation of planar magnetic films on MEMS chips for *in situ* TEM studies.

In this context, the preparation of a planar FeRh thin film on a MEMS-based heating chip using a focused ion beam (FIB) secondary electron microscope (SEM) is presented. The use of a SEM stub with 45° faces, custom-designed by DENSsolutions®, provides a new and innovative method for the transfer and preparation of planar magnetic thin films for the purpose of *in situ* TEM investigations.

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2. Experimental

A thin film of ordered α' -FeRh alloy (~ 55 nm thick) was grown epitaxially on a clean (001) MgO substrate (~ 500 μm thick) by conventional DC magnetron sputter co-deposition, as described in [6]. The sample was fixed onto a glass slide (FeRh film face-down) using Kemdent® wax and cut into ~ 1.5 mm square sections using a circular diamond saw. The glass slide was then placed on a hot plate to melt the wax and release the square sections, which were cleaned with acetone to remove any waxy residue. A dried square section was then fixed, again with FeRh film face-down, onto a Pyrex® specimen stub using ethyl cyanoacrylate (Loctite® ‘super glue’) and cured at 80°C for 2 hours. It was then mounted onto a tripod polisher and thinned to ~ 5 μm with a Struers® Labopol wheel polisher and diamond paper, using progressively finer grades (30 – 1 μm grit). An Omniprobe® Cu grid was then overlaid onto the sample and fixed using an epoxy adhesive (Araldite®) and cured at 80°C for 12 h. The stub / sample / Cu-grid combination was then immersed fully in acetone for 2 hours to dissolve the ‘super glue’ and release the remaining thinned sample / Cu-grid combination. The sample was then inserted in a FEI Dual Beam FIB Nova 200 for transfer to a DENSsolutions® MEMS-based heating chip (mounted on the custom SEM stub with 45° faces), as well as final thinning of the FeRh planar sample for the purpose of *in situ* TEM investigation. The FIB instrument comprises a 30 kV electron (e^-) column for SEM and a 30 kV sidewinder ion column (Ga^+), mounted at 52° to the e^- column. The system is equipped with an *in situ* micro-manipulator and a gas injector for the *in situ* deposition of Pt. Magnetic imaging was performed using a JEOL ARM-200cF TEM in Lorentz mode and Fresnel fringes in over- or under-focus provides contrast from in-plane DWs.

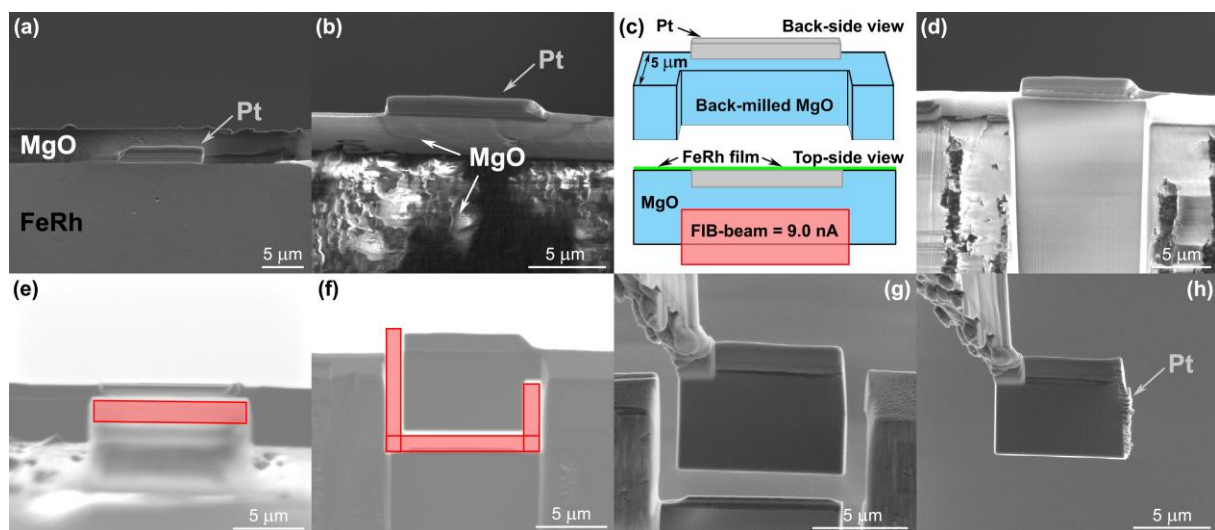


Figure 1 (a,b) SEM images showing (a) front-; and (b) back-side views of the FeRh thin film, MgO substrate and protective Pt layer. (c) Schematics of the back- and top-side views of the FIB-milling of the substrate using a current of 9.0 nA (red boxed region). (d) SEM image of the FIB back-milling shown in (c). (e,f) FIB images showing the (e) top-side view of back-milling; and (f) undercutting of the lamella (boxed regions), both using a current of 0.9 nA. (g,h) SEM images of (g) the welding of the micro-manipulator to the lamella and its release from the bulk sample; and (h) deposition of the Pt layer to allow for its attachment to the MEMS heating chip.

3. Results

This section illustrates our hybrid mechanical and FIB-based polishing approach for the preparation of an electron-transparent planar thin film FeRh sample on a MEMS heater chip, for the purpose of *in situ* TEM investigations.

Fig. 1 presents the initial milling and release of a thick TEM lamella, with dimensions of ~ 9 $\mu\text{m} \times 5$ $\mu\text{m} \times 1$ μm , from the polished sample. The sample was tilted until the e^- beam was parallel to the face of the FeRh film and the angle was noted as 6° . The SEM image of Fig. 1a shows the FeRh thin film grown on the MgO substrate and a protective Pt layer. The Pt was deposited using the FIB (30.0 kV) at a current of 0.93 pA and at an angle of 10° away from the face of the FeRh film to prevent

unwanted Pt deposition on its surface. The sample was then rotated 180° to ensure the FeRh film undergoes limited exposure to both the e^- beam and FIB (Fig. 1b), as well as a favourable orientation for transfer to the heating chip. The sample was then tilted so that the face of the FeRh film (was positioned 1° away from the ion-beam path and back-milling of the MgO substrate was performed at a current of 9.0 nA (Fig. 1c). The FIB milling was monitored using the e^- beam until the milled section reached a sufficient depth (Fig. 1d). Back-milling continued using progressively lower currents of 2.7 nA and 0.9 nA until a lamella thickness of $1\mu\text{m}$ was achieved (Fig. 1e). The sample was then tilted to 0° , rotated by 180° and three undercuts were made simultaneously (Fig. 1f), also using a FIB current of 0.9 nA, to free most of the lamella from the polished sample (Fig. 1g). The sample was rotated by 180° , tilted back to 1° away from the FIB path and both the micro-manipulator and gas injector were inserted. The micro-manipulator was brought into contact with the top left corner of the lamella and welded together with Pt using the e-beam (5.0 kV, 1.6 nA). The FIB was then used to release the lamella from sample using a current of 0.9 nA, as well as deposit a layer of Pt on the right-hand side of the lamella ($I = 90\text{ pA}$) to allow for its attachment to the MEMS heating chip (Fig. 1h).

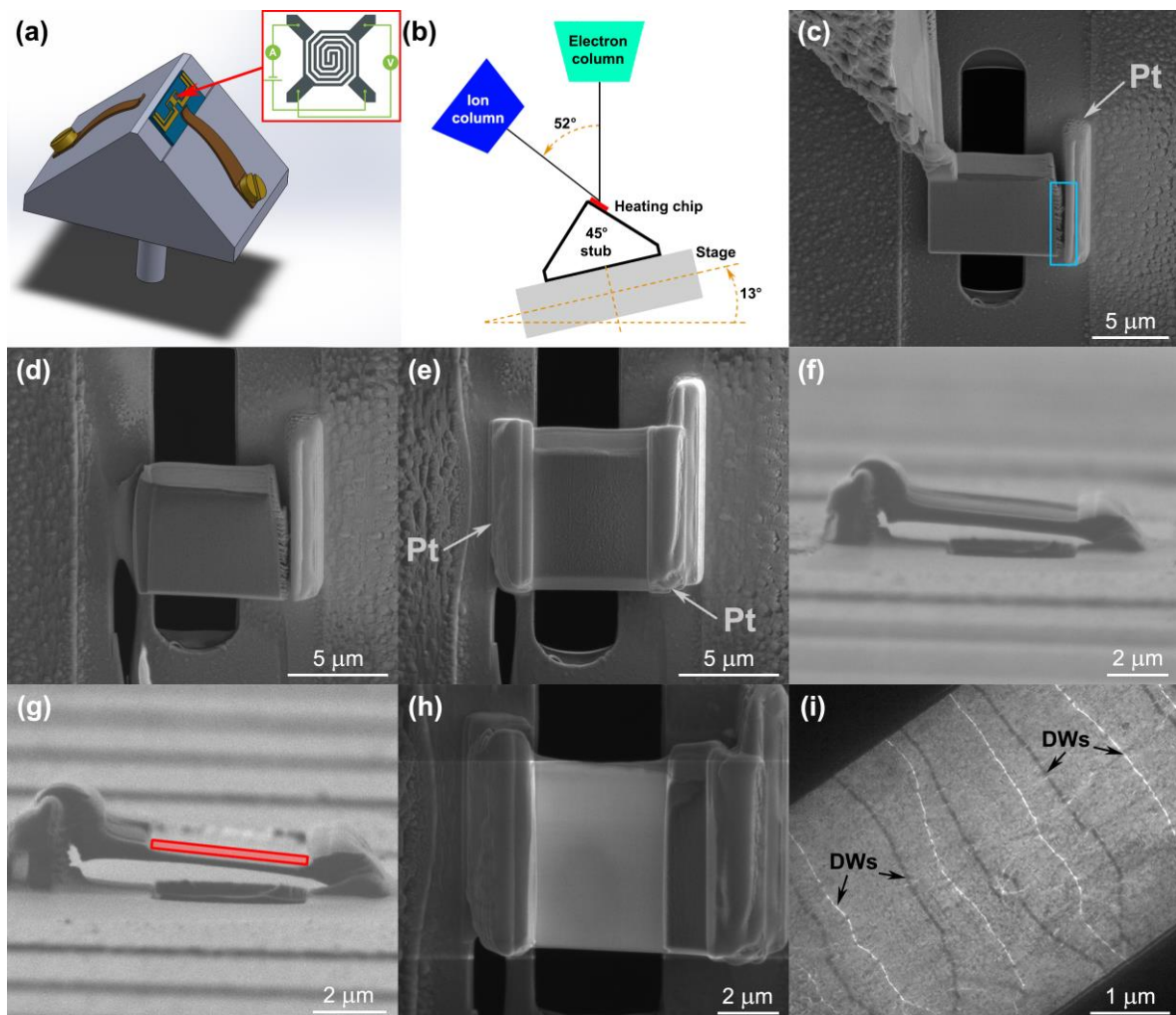


Figure 2 (a) 3D model showing the positioning of the MEMS chip and heating coil (inset), on the custom-designed SEM stub with 45° faces. (b) Schematic diagram illustrating the configuration of the 45° stub / heating chip in relation to the e^- and FIB columns within the FEI Helios workstation. (c-e) SEM images of the lamella (c) brought into contact with the pre-deposited Pt step (boxed region); (d) welded to the Pt step with the e^- beam and released from the micro-manipulator; and (e) reinforced with additional Pt deposition. (f,g) FIB images showing the side-view and thinning of the lamella using a current of (f) 0.26 nA; and (g) 90 pA, as denoted by the red boxed region in (g). (h) SEM image of the electron-transparent planar FeRh thin film. (i) Fresnel TEM image showing the presence of DWs in the FeRh thin film at $T = 200^\circ\text{C}$, in the ferromagnetic state.

Fig. 2 presents the transfer of the TEM lamella onto the MEMS heating chip and final FIB milling into an electron-transparent FeRh thin film. The 3D model of Fig. 2a shows how the MEMS-based heating chip, incorporating a heating coil (Fig. 2a, inset), is positioned on the 45°-faced SEM stub. Fig. 2b illustrates the configuration of the 45° stub / heating chip in relation to the e^- and FIB columns, to allow for placement of lamella on the heating chip. The tilting of the stage to 13° provides a 7° difference in angle between the ion column and heating chip surface, which allows an effective milling path for the ion beam without damaging the heating element beyond the lamella. The micro-manipulator brings the Pt layer on right-hand side of the lamella in contact with a pre-deposited Pt step ($\sim 10 \mu\text{m} \times 1 \mu\text{m} \times 1 \mu\text{m}$) (Fig. 2c). SEM imaging is then magnified to 500kx and rastered along the Pt-Pt interface for 5 min using the e^- beam shifts to weakly weld the surfaces together (boxed region). The FIB is then used to release the lamella from the micro-manipulator (Fig. 2d) ($I = 0.9 \text{ nA}$) and, after tilting to 45°, deposit additional Pt to firmly weld both sides of the lamella to the heating chip (Fig. 2e) ($I = 90 \text{ pA}$). The stage is tilted back to 13° and 12° for final milling using progressively lower currents of 0.26 nA (Fig. 2f) and 90 pA (Fig. 2g), respectively, until the lamella is $\sim 100 \text{ nm}$ thick. A final low energy FIB polish was carried out at 5.0 keV and 47 pA at an incidence angle of +7° (stage tilt of 20°), until the SEM image revealed a clean planar TEM lamella with an electron-transparent area of $\sim 6 \mu\text{m} \times 4 \mu\text{m}$ (Fig. 2h). *In situ* heating to 200°C within the TEM in Lorentz mode (Fig. 2i) revealed the presence of DWs in the planar FeRh thin film, visible as black and white lines (arrowed).

4. Discussion

This study has provided a simple methodology for the preparation of large planar magnetic thin films on MEMS-based chips for the purpose of *in situ* TEM investigations. The SEM images of Fig. 1a,b confirm the preliminary polishing as an effective approach to reduce the MgO substrate thickness from $\sim 500 \mu\text{m}$ to $\sim 5 \mu\text{m}$, before insertion into the FIB SEM for the main preparation. The subsequent FIB back-milling using gradually lower currents of 9.0 nA, 2.7 nA and 0.9 nA, whilst monitoring the progress using the e^- column, offered excellent control over the milling-rate and thickness of the lamella section as it approached 1 μm , before being undercut and transferred (Fig. 1f,g).

It is evident the 45°-faced SEM stub provides a favourable geometrical arrangement for the near-parallel placement of the lamella on the heating chip, with the magnetic thin film face-down away from exposure of the e^- beam and FIB. One unconventional caveat of this setup is the need to use the e^- beam for both Pt deposition and welding Pt interfaces together. This is due to the FIB not having the necessary access to the point of contact between the lamella and micro-manipulator, where the e^- beam supplied an adequate Pt weld instead (Fig. 1g). In addition, the gas injector collides with the heating chip when on the 45° face and so the e^- beam was rastered along the interface of the Pt on the right-hand side of the lamella and the Pt step to provide a temporary weld whilst the micro-manipulator was removed. Nevertheless, the lamella was transferred and welded onto the heating chip successfully to enable FIB back-milling of the MgO substrate into an electron-transparent planar FeRh thin film. This was confirmed through TEM investigation in Lorentz mode, where the sample was heated to 200°C *in situ* using the MEMS chip and Fresnel fringes were clearly visible, indicating the presence of DWs.

In summary, a planar FeRh thin film sample was prepared on a MEMS-based TEM heating chip using a novel 45°-faced SEM stub. TEM investigation revealed the presence of DWs and confirms the applicability of this method for preparing planar samples for *in situ* TEM studies.

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Acknowledgements

The authors thank the EPSRC for funding (grants EP/M019020/1 & EP/M018504/1).